

PATENT ABSTRACTS OF JAPAN

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(21)Application number : 03-205308 (71)Applicant : CHLORINE ENG CORP LTD

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(54) METHOD FOR PURIFYING SILICONE OIL

(57)Abstract:

PURPOSE: To efficiently purify a silicone oil with reduced cost by extracting low molecular weight substances from a silicone oil containing methylphenylsiloxane.

CONSTITUTION: A silicone oil is purified by bringing a phenylsilicone oil comprised of a copolymer comprised of methylphenylsiloxane units of the formula: $-\text{Si}(\text{CH}_3)(\text{Ph})\text{O}-$ (I) (wherein Ph represents a phenyl group) and dimethylsiloxane units of the formula: $-\text{Si}(\text{CH}_3)_2\text{O}-$ (II) wherein the methylphenylsiloxane unit content of all the siloxane units is 50% or less into contact with carbon dioxide in a subcritical or supercritical state so as to extract off low molecular weight substances from the silicone oil.

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DETAILED DESCRIPTION

[Detailed Description of the Invention]

[0001]

[Industrial Application] This invention relates to the silicone oil (henceforth "methylphenyl silicone oil") purification approach which consists of a copolymer which consists of a dimethylsiloxane unit and a methylphenyl siloxane unit, and relates to the purification approach of this methylphenyl silicone oil which becomes a detail from carrying out extraction separation of the low-molecular-weight matter with which the content of a methylphenyl siloxane unit is contained in it from 50% or less of methylphenyl silicone oil with the carbon dioxide of subcritical or a supercritical condition more.

[0002]

[A technical background] Although general silicone oil consists of a homopolymer of dimethylsiloxane, the methylphenyl siloxane system silicone oil containing a methylphenyl siloxane is used for the application which needs thermal resistance, cold resistance, etc. Since the low-molecular-weight matter which is mainly an unreacted monomer and oligomer is contained in these silicone oil after manufacture by the polymerization of a siloxane and degradation is invited to it, it is necessary to remove these. Although these are removed from the former by the usual vacuum distillation, a molecular distillation method, etc., in order to attain clearance of the desired low-molecular-weight matter, very severe conditions must be maintained strictly for a long time, and, moreover, it is necessary to repeat such vacuum distillation, molecular distillation, etc. usually two or more times. Therefore, the approach the actual condition is that purification of the silicone oil by vacuum distillation or molecular distillation has also required costs with technical difficulty, and it can remove efficiently the low-molecular-weight matter in these silicone oil cheaply is searched for.

[0003] On the other hand, the supercritical extraction which used the carbon dioxide (carbon dioxide gas) is known from Men, such as a fluid of a supercritical condition especially safety, and profitability, as the extract approach of a minute amount organic compound, and it is used for purification of the matter or the extract of the useful matter. Although it was possible for the low-molecular-weight matter in the silicone oil which consists of a dimethylsiloxane homopolymer to have dissolved in the carbon dioxide of a supercritical condition about the behavior of the silicone oil to the carbon dioxide of this supercritical condition, therefore to have extracted and refined only the low-molecular-weight matter, the low-molecular-weight matter in the silicone oil which consists of a methylphenyl siloxane homopolymer did not dissolve in the carbon dioxide of a supercritical condition at all, but it was known that the purification by supercritical extraction is impossible.

[0004]

[The technical problem which should solve invention] Therefore, the object of this invention is offering the approach the low-molecular-weight matter's being efficiently extracted from the silicone oil containing a methylphenyl siloxane, and silicone oil's being refined.

[0005]

[Means for Solving the Problem] Since the low-molecular matter in the methylphenyl silicone oil which consists of a methylphenyl siloxane homopolymer did not dissolve in the carbon dioxide of a

supercritical condition at all as above-mentioned, it was thought that purification of the methylphenyl silicone oil by supercritical extraction was impossible.

[0006] However, it is almost the case which the silicone oil containing the methylphenyl siloxane currently actually used on industry does not consist of a methylphenyl siloxane homopolymer, and consists of a copolymer containing dimethylsiloxane. Then, this invention person etc. completed this invention, as a result of inquiring wholeheartedly from a viewpoint that supercritical extraction may be able to refine depending on the content of a methylphenyl siloxane, even if it was the silicone oil containing a methylphenyl siloxane.

[0007] This invention is the content (content of the methylphenyl siloxane unit over all siloxane units.) of a methylphenyl siloxane, even if it is the silicone oil containing a methylphenyl siloxane. It is called "phenyl content" below. The low-molecular-weight matter contained in it may dissolve in the carbon dioxide of a supercritical condition, and it is based on having found out that the solubility of extent which can be used for purification of the methylphenyl silicone oil concerned was accepted.

[0008] That is, as shown in a postscript, as a result of trying the extract by the carbon dioxide of a supercritical condition in various content, when it was the polyphenyl methyl siloxane of 30% or less of phenyl group content preferably, it was found out about 50% or less that clearance of the low-molecular-weight matter needed practically can attain. Therefore, this invention is Formula I.;

- -Si(CH₃) (Ph)-O-- (I) (Ph expresses a phenyl group among a formula.) The methylphenyl siloxane unit expressed and formula II;

- -Si(CH₃)₂-O-- It is the purification approach of silicone oil that consist of a copolymer which consists of dimethylsiloxane units expressed with (II), and the content of the methylphenyl siloxane unit over the total siloxane number of unit consists of making 50% or less of phenyl silicone oil contact the carbon dioxide of subcritical or a supercritical condition, and carrying out extraction separation of the low-molecular-weight matter in this silicone oil.

[0009] The low-molecular-weight matter removed by this invention approach means the oligomer which consists of units, such as an unreacted monomer of a methylphenyl siloxane and dimethylsiloxane, and it to about ten pieces. Moreover, especially the molecular weight of the polysiloxane which constitutes the silicone oil set as the object of purification by this invention approach is usually abbreviation on an average, although not limited. It is preferably applicable about the silicone oil which consists of a polysiloxane including about 400 siloxane unit.

[0010] The critical points of a carbon dioxide are about 31.0 degrees C and 75.3kg/cm², and it is referred to as being in a supercritical condition to be in the condition of having exceeded this and to have the in-between property of a liquid and a gas, i.e., the consistency near a liquid, and a diffusion coefficient near a gas. Moreover, although there is no clear definition, generally a subcritical state exists near the supercritical condition, is about 50kg/cm² or more in the temperature of about 25 degrees C or more, and pressure in a carbon dioxide, is in the above-mentioned critical temperature or below a pressure, and says what has the in-between property of the above liquids and a gas.

[0011] When the conditions used for the usual supercritical extraction and a subcritical extract can be used in operation of this invention approach and 80 - 500 kg/cm², 70 to 120 degree C, and a subcritical extract are usually included in the case of supercritical extraction, it is desirable to carry out using the carbon dioxide in a supercritical condition or a subcritical state under 50 - 500 kg/cm² and conditions of about 25-120 degrees C. Moreover, the equipment used for the usual supercritical extraction and a subcritical extract can be used for the equipment for enforcing this invention approach. this invention approach can be performed according to the process flow shown in drawing 1. In drawing 1, a carbon dioxide is supplied to a compressor 3 via a heat exchanger 2 from the carbon-dioxide cylinder 1, and it compresses to a predetermined pressure, is further made extract temperature predetermined by the heat exchanger 4, it is made supercritical or a subcritical state, and introduces to the extract tub 5. The extract tub 5 adjusted to predetermined temperature is filled up with the silicone oil which should be refined, the carbon dioxide containing the low-molecular-weight matter after extracting is decompressed through a reducing valve 6, it introduces into the separation tub 7, and the low-molecular-weight matter is separated. The carbon dioxide with which the low-molecular-weight matter was separated is recycled

via a heat exchanger 2.

[0012]

[Effect of the Invention] This invention extracts the low-molecular-weight matter from the silicone oil containing a methylphenyl siloxane, and the method of refining silicone oil cheaply efficiently is offered.

[0013]

[Example] Hereafter, an example explains this invention further.

Example 1 bore of 30mm, container made from a stainless steel with a die length of 11cm (it is thickness to an upper bed and a soffit thing equipped with porous sheet [made from 2mm titanium] (10 micrometers of diameters of pore)) (viscosity abbreviation 540 cps (25 degrees C) --) from -- the becoming extract tub -- methylphenyl silicone oil of 10 g Average molecular weight 4900 [about], about 30% of phenyl content, about 13 % of the weight of oligomer content It is filled up. Carbon dioxide of the supercritical condition of 80 degrees C and 350 kg/cm² It supplied upwards for about 8 minutes from the container soffit at the rate of 6 l/min, the low-molecular-weight matter was extracted, and uptake of about 1.5g low-molecular-weight matter was carried out to the extract tub.

[0014] the silicone oil obtained after extract purification -- GPC (GelPermeation Chromatography) when it analyzes, not being contained substantially shows the low-molecular-weight matter -- having -- moreover, the viscosity -- 540 cps before processing (25 **) from -- 620 cps (25 **) It was going up and the effectiveness of low-molecular-weight matter clearance was accepted.

As silicone oil which should be carried out the product made from example dispermy, it is viscosity abbreviation. 110 cps (25 degrees C), Methylphenyl silicone oil of a mean molecular weight 2500 [about], about 10% of phenyl content, and about 15 % of the weight of oligomer content is used. It is the rate of flow about the carbon dioxide of the supercritical condition of 80 degrees C and 300 kg/cm². Except having supplied for about 10 minutes by 5 l/min, silicone oil was refined by the same approach as an example 1, and uptake of about 1.5g low-molecular-weight matter was carried out to the extract tub.

[0015] it shows that the low-molecular-weight matter is not substantially contained by the GPC analysis of the silicone oil obtained after extract purification -- having -- moreover, the viscosity -- 110 cps before processing (25 **) from -- 130 cps (25 **) It went up and the effectiveness of low-molecular-weight matter clearance was accepted.

As silicone oil which should be refined example 3, it is viscosity abbreviation. Methylphenyl silicone oil of 480 cps (25 degrees C), a mean molecular weight 4100 [about], about 25% of phenyl content, and about 11 % of the weight of oligomer content is used, and it is the rate of flow about the carbon dioxide of the supercritical condition of 80 degrees C and 350 kg/cm². Silicone oil was refined by the same approach as an example 1 except having supplied for about 10 minutes by 6 l/min.

[0016] it shows that the low-molecular-weight matter is not substantially contained by the GPC analysis of the silicone oil obtained after extract purification -- having -- moreover, the viscosity -- 480 cps before processing (25 **) from -- 510 cps (25 **) It went up and the effectiveness of low-molecular-weight matter clearance was accepted.

As silicone oil which should be refined example 4, it is viscosity abbreviation. Methylphenyl silicone oil of 530 cps (25 degrees C), a mean molecular weight 4900 [about], about 30% of phenyl content, and about 10 % of the weight of oligomer content is used, and it is the rate of flow about the carbon dioxide of the supercritical condition of 80 degrees C and 350 kg/cm². Silicone oil was refined by the same approach as an example 1 except having supplied for about 10 minutes by 8 l/min.

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As silicone oil which should be refined example 5, it is viscosity abbreviation. 650 cps (25 degrees C), a mean molecular weight 7400 [about], about 40% of phenyl content, oligomer content abbreviation 9% of the weight of methylphenyl silicone oil is used, and it is the rate of flow about the carbon dioxide of

the supercritical condition of 70 degrees C and 350 kg/cm². Silicone oil was refined by the same approach as an example 1 except having supplied for about 10 minutes by 9 l/min.

[0018] it shows that the low-molecular-weight matter is not substantially contained by the GPC analysis of the silicone oil obtained after extract purification -- having -- moreover, the viscosity -- 650 cps before processing (25 **) from -- 730 cps (25 **) It went up and the effectiveness of low-molecular-weight matter clearance was accepted.

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As silicone oil which should be refined example of comparison 1, methylphenyl silicone oil of about 1100 viscosity cps (25 degrees C), a mean molecular weight 7100 [about], about 52% of phenyl content, and about 10 % of the weight of oligomer content was used, and silicone oil was refined by the same approach as an example 1 except having supplied the carbon dioxide of the supercritical condition of 80 degrees C and 350 kg/cm² for about 10 minutes by rate-of-flow 10 l/min.

[0020] although it was shown that the low-molecular-weight matter is contained a little by the GPC analysis of the silicone oil obtained after extract purification -- the viscosity -- 1100 cps before processing (25 degrees C) from -- 1300 cps (25 degrees C) It went up and the effectiveness of low-molecular-weight matter clearance was accepted.

As silicone oil which should be carried out the product made from the example dispermy of a comparison, methylphenyl silicone oil of about 2500 viscosity cps (25 degrees C), average molecular weight 15800 [about], about 56.6% of phenyl content, and about 11 % of the weight of oligomer content is used, and it is the rate of flow about the carbon dioxide of the supercritical condition of 70 degrees C and 350 kg/cm². Silicone oil was refined by the same approach as an example 1 except having supplied for about 20 minutes by 8 l/min. Uptake of about 0.5g low-molecular-weight matter was carried out to the extract tub.

[0021] it shows that the low-molecular-weight matter is contained by the GPC analysis of the silicone oil obtained after extract purification -- having -- moreover, the viscosity -- 2500cps before processing (25 **) from -- it hardly changed and the effectiveness of low-molecular-weight matter clearance was not accepted.

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- -Si(CH₃) (Ph)-O-- (I) (Ph expresses a phenyl group among a formula.) The methylphenyl siloxane unit expressed and formula II;

- -Si(CH₃)₂-O-- It is the purification approach of silicone oil that consist of a copolymer which consists of dimethylsiloxane units expressed with (II), and the content of the methylphenyl siloxane unit over the total siloxane number of unit consists of making 50% or less of phenyl silicone oil contact the carbon dioxide of subcritical or a supercritical condition, and carrying out extraction separation of the low-molecular-weight matter in this silicone oil.

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CLAIMS

[Claim(s)]

[Claim 1] Formula I;

- -Si(CH₃) (Ph)-O-- (I) (Ph expresses a phenyl group among a formula.) The methylphenyl siloxane unit expressed and formula II;

- -Si(CH₃)₂-O-- The purification approach of silicone oil that consist of a copolymer which consists of dimethylsiloxane units expressed with (II), and the content of the methylphenyl siloxane unit over the total siloxane number of unit consists of making 50% or less of phenyl silicone oil contact the carbon dioxide of subcritical or a supercritical condition, and carrying out extraction separation of the low-molecular-weight matter in this silicone oil.

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